A 8 DATA REVIEW FOR ORGANIC ANALYSES

A 8.1 Introduction

Every analysis has certain analytical activities that are used as "indicators" of performance of the analytical system, generally referred to as "quality control" (QC). The results of each of the QC activities must be reviewed in order to determine the potential impact that may be reflected upon the associated data. While the following procedural outline is intended to promote consistency in actions resulting from quality control data, a thorough review must include professional judgement in the assessment and determination of appropriate corrective action(s). It may very well be appropriate to take a path different from the guidance below, however, this should be done in consultation with lead analysts, supervisors and the Branch Quality Assurance Officer. Thorough documentation must be included in the project file concerning the rationale for the deviation. The quality of the data resulting from such a review is communicated to the users of the data by a formal system of data qualifiers. [See Chapter 5, Section 5.6 for a complete listing of the data qualifiers with definitions.]

A 8.2 Holding Time

A 8.2.1 Objective. Holding time for any analysis is defined as the "technical holding time" or actual age of the sample, that is, the amount of time lapsed between the time the sample was taken and the time the sample was analyzed or prepared for analysis. Some methods have holding times prior to sample prep/extraction and another period of time for analysis of the extracts or prep solutions.

A 8.2.2 Criteria. Technical holding time criteria are listed in the LOQAM for the various organic analyses. For those matrices that do not have established criteria, professional judgement must be used based on the best available information and criteria available for similar matrices. In these instances consult the Branch QAO or Supervisor for guidance.

A 8.2.3 Evaluation. Review the samples and extracts for the elapsed time in days for those analyses that have holding times defined in days and in hours for those analyses that have holding times defined in hours. Be sure to account for the differences in holding times for any samples that were not preserved. For analyses that have a preparation/extraction step holding times for each segment of the analysis must be evaluated. If any segment of the holding time is exceeded (i.e., time lapsed prior to extraction or time lapsed prior to analysis of the extract) then for purposes of this evaluation the holding time for that sample is considered

to be exceeded.

A 8.2.4 Action. For each sample that exceeds the holding time flag all analytes with a "J" flag qualifier and include the footnote that holding times were exceeded. If for any reason the exceeded holding time is such that there is uncertainty for the validity of the qualitative analysis, negative results should be flagged "R" and positive hits flagged as "J".

A 8.3 Method Blanks

A 8.3.1 Objective. A method blank is used to assess the associated samples in a specific preparation batch for possible contamination during the preparation and analysis procedure. It generally consists of a sample matrix similar to the batch of associated samples (when available) that is known to be free from the analytes of interest, shall be processed simultaneously with, including all reagents that are used on the samples, and under identical conditions as samples through all steps of the analytical procedure. *Note: Field blanks, trip blanks, and field equipment rinse blanks are used to determine potential contamination by field operations and are used by the sampling organizations for their internal quality control activities. These samples should be treated and reported to project leaders as any other sample. ASB makes no analytical evaluation on data reporting based on contamination of field QC samples.*

A 8.3.2 Criteria. The goal of all method blanks is to have no contamination. The method blank shall be analyzed at a minimum of 1 per preparation batch. For those instances (such as volatile organics analyses in water) for which no separate preparation method is used the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

A 8.3.3 Evaluation. Target analyte contaminants in the blank are compared to the associated samples and action taken as described below. Non-target analyte contaminants must be reviewed for their potential interference with the analysis as well.

A 8.3.4 Action. The primary corrective action for contaminated blanks is to correct the problem and reanalyze all affected samples. However, in consideration of project objectives and the specific impact of the contamination, it may be appropriate to use the data. In each instance of blank contamination all possible and practical steps must be taken to correct or minimize the problem. A target analyte found in a blank and also found in an associated sample may be

considered for reporting when present at a ratio of at least 10/1, sample to blank. Notably, there are some specific components of special consideration (listed below) that are exceptions to this ratio. Blank values are not subtracted from reportable results unless directed by the method. For those analyses for which the associated blank has contamination, and it is determined that the data must be reported from that run, use the following guidance:

Target compounds below the 10/1 sample/blank ratio shall be reported in samples as follows:

If the sample result is less than the MQL, report non-detect at the normal sample MQL:

Example:	MQL = 10 U	MQL = 10 U
	blank = 6	blank = 30
	sample = 3	sample = 5
	report = 10 U	report = 10 U

If the sample result is greater than the MQL, round the sample value up to the next highest 1 significant figure and report with the U flag.

Example:	MQL = 10 U	MQL = 0.5 U	MQL = 5.0 U
	blank = 9.0	blank = 0.55	blank = 12
	sample = 22	sample = 0.62	sample = 9.2
	report = 30 U	report = 0.7 U	report = 10 U

A 8.3.4.1 Non-target contaminants must likewise be evaluated for impact on the results. They may or may not have a direct impact on the results. Project data quality objectives need to be considered in the reporting of data for non-target contamination or in further corrective action taken on the re-analysis of associated samples. It must be noted that non-target contaminants that do not directly interfere with a specific analysis should always be considered an issue since their very presence is indicative of an undesired intrusion of components and efforts must be made to minimize or eliminate the problem. Supervisors and the Branch QAO shall be consulted for guidance.

A 8.3.4.2 Special Consideration Volatiles Contamination from some volatile components are more common and should not be considered for reporting until a ratio of 10/1 sample to blank is reached. This includes methylethylketone and acetone. These compounds may be reported following the above procedure using the 10/1 ratio. Notably, there may be times for which professional judgement dictates a different approach to

reporting these components. The reporting rationale, if different from this procedure, shall be documented.

A 8.3.4.3 Special Consideration Semivolatiles: Contamination from some semivolatile components are more common and should not be considered for reporting until a ratio of 10/1 sample to blank is reached. These include bis(2-ethylhexyl) phthlate, diethyl phthalate, dibutyl phthlate, diethyl phthalate, n-nitrosodiphenylamine, silicones, octadecenamide, and phthalic acid. These compounds may be reported following above procedure using the 10/1 ratio as the reporting level. Notably, there may be times for which professional judgement dictates a different approach to reporting these components. The reporting rationale, if different from this procedure, shall be documented.

A 8.3.4.3.1 Butoxyethoxyethanol and similar compounds are known to be common contaminants of tubing used in sampling equipment. It often occurs, however, that the analytical blanks do not contain the contaminant and samples and field blanks/rinsate blanks do. It is important that the compounds are reported as would any other "field contaminants" in order for the sampling organizations to be made aware of this issue. It is appropriate as well to contact the project leader to alert him/her of the problem.

A 8.3.4.3.2 Extraction artifacts of chlorinated water prepared with methylene chloride should not be reported. These include chlorinated cyclohexenes, cyclohexanes, and cyclohexanols. If these components are present in a sample the reviewer must investigate the presence of chlorine with sampling personnel and/or other sources.

A 8.4 Calibration - Initial (ICAL)

A 8.4.1 Objective. Before samples are analyzed, all instrumentation must have an initial standardization using calibration standards. Most often this initial calibration consists of a calibration curve over a concentration range as specified by the method, by SOPs or by the LOQAM. Additionally, the accuracy of the standards used for the calibration must be verified by a secondary "check standard" obtained from a source different from the initial calibration standard. Also, each time a fresh stock standard is prepared the resulting working standards are checked against the previous stock as a cross check.

A 8.4.2 Criteria. Specific criteria for the multipoint calibrations are listed in the methods and/or the ASB LOQAM, Chapter 8. These established criteria must be met for analyses to proceed. Additionally, criteria must be met for comparison to the check standard and the cross check analysis to the previous stock standard . Special and rare circumstances may indicate in the professional opinion of the Lead Analyst and the Section Chief that analyses should proceed even with criteria outside the established limits. If analyses continue using an ICAL that does not meet criteria there must be complete documentation of the process decision that includes all details and clear justification for the action.

A 8.4.3 Evaluation Insure that the initial calibration was performed according to established methods or operational procedures and that it meets the established criteria. If the acceptance criteria were not met and analyses did proceed, insure that the appropriate documentation is in place for details and justification of the process.

A 8.4.4 Action. For any individual components that were outside of the established criteria add the J qualifier flag to the results. If, for any reason, the ICAL indicates that any specific component has performed so poorly that the qualitative analysis for that individual component is in question, and there was a decision to proceed, the data report shall reflect the notation of the specific compound flagged as R (rejected).

A 8.5 Calibration Check Standard

A 8.5.1 Objective. The calibration check standard consists of a standard material used to verify the accuracy of the initial calibration standards. The standard used for the calibration check should be purchased from a different vendor than the initial calibration standard or, if from the same vendor, must be from a different lot number. The calibration check standard must be analyzed each time a new calibration curve is generated using a new stock standard stock and must be run at a minimum of once each quarter as a stability check.

A 8.5.2 Criteria. Criteria for an acceptable comparison are: 75% of the components must have less than or equal to 10% difference. If any one component has a % difference greater than 20% the system must be investigated, problems corrected and the standards analysis repeated. The same criteria shall be applied for the check standard and the cross check to the previous stock analysis. If it is the professional judgement of the lead analysts and the Section Chief that analyses should proceed (concurrence of both is required) with criteria not met,

the details and justification of the decision to proceed shall be completely and clearly documented.

A 8.5.3 Evaluation. Insure that the calibration standards have been appropriately verified by the check standard and are within acceptable limits. If it was the professional judgement of the lead analysts and the Section Chief, that analyses proceeded with criteria not met, insure that complete documentation of the details and justification of the decision to proceed is included.

A 8.5.4 Action. Investigate the problem, take corrective action, and recalibrate and check. If there is a decision to proceed with unacceptable results, insure that complete documentation of the details and justification of the decision to proceed is included If the decision is to proceed flag the data as follows: For each individual component outside of the established criteria add the J qualifier flag to the results. If, for any reason, the calibration check indicates that a specific analyte has performed so poorly that the analysis is in question and there was a decision to proceed, flag all affected compounds with the "R" flag.

A 8.6 Calibration - Continuing Verification (CCV)

A 8.6.1 Objective. After an acceptable initial calibration curve has been established it is necessary to verify with a standard at a concentration of approximately the mid point of the calibration curve as described by SOPs, required methods, or the LOQAM. This standard is called the continuing calibration verification (CCV). Once the ICAL curve has been verified as acceptable by a check standard the CCV is run at specified intervals to monitor the stability of the standards and of the instrumental system.

A 8.6.2 Criteria. The CCV should be analyzed at specified intervals and meet acceptance criteria as defined by the individual methods, SOPs or the LOQAM. Once the CCV is determined as acceptable, analyses may proceed. If the CCV does not meet criteria special circumstances may dictate by professional opinion of the Lead Analyst and the Section Chief that analyses should proceed. If the decision is to proceed using a CCV that does not meet criteria there must be complete documentation of the process with details and justification of the decision to proceed.

A 8.6.3 Evaluation. Insure that the CCV was analyzed at appropriate intervals and that criteria were met as specified by the method, SOP or LOQAM. If the acceptance criteria were not met and analyses did proceed, insure that the appropriate documentation is in place for details and justification of the process.

A 8.6.4 Action. Investigate the problem, correct any problems found and re-analyze the CCV or it may be determined necessary to rerun the complete ICAL. For all individual components that were outside of the established criteria add the J qualifier flag to the results. If, for any reason, the CCV indicates that any specific component has performed so poorly that the qualitative analysis is in question and there was a decision to proceed with the analysis, insure that the report reflects an appropriate notation of the specific compound flagged as R (rejected).

A 8.7 Calculating/Reporting of Target Analytes

A 8.7.1 Objective. The objective of all analyses is to perform calculations of sample results within the established working linear range of the instrumental system as established by the calibration curve.

A 8.7.2 Criteria. Each target analyte determined to be present in the sample must be calculated within the established linear range of the system as defined by the calibration curve. Target analytes above the highest concentration of the standard curve require dilutions where possible to bring them into the linear range, best accomplished when diluted into the mid-level of the established curve. To provide the most complete analysis, some samples may require several analyses, injecting the least possible dilution (or no dilution) to determine low concentrations and injecting very dilute to bring high concentrations within the linear range. Target analytes determined to be present below the established minimum quantitation limit are reported as described below.

A 8.7.3 Evaluation. Insure that all target analytes reported above the minimum quantitation limit were calculated within the linear range as established by the calibration curve. Insure that any target analytes reported below the minimum quantitation limits met all qualitative criteria for reporting of a positive hit.

A 8.7.4 Action. If all target analytes above the minimum quantitation limit were within the linear range as established by the calibration curve no flags are necessary. Any outside the curve require the “J” qualifier flag. All analytes reported as present below the minimum quantitation limit must have the “J” qualifier flag.

A 8.8 Laboratory Control Sample (LCS) - Defining Method Precision and Bias

A 8.8.1 Objective. The LCS is used to evaluate the performance of the total laboratory system and is processed through all steps of the method for preparation

and analysis. Establishing acceptance with the LCS defines that the method was in control for both precision and bias as dictated by acceptance criteria for % RSD from either historical data or from method requirements.

A 8.8.2 Criteria. An LCS must be analyzed at a minimum of 1 per preparation batch. In those instances for which no separate preparation method is used (e.g., volatiles in water) the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples. The matrix to be spiked should be controlled and known to be free of analytes of interest or alternatively the LCS may be a reference matrix of known and verified concentrations of analytes. The LCS spike components should be as specified by test methods or specific project needs. If there are no specified components the laboratory shall spike per the following:

A 8.8.2.1 Pesticides/PCBs For those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene, and PCBs, and for compounds with co-eluting peaks, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported. However, the laboratory shall insure that all targeted components are included in the spike mixture over a 2 year period.

A 8.8.2.2 Extractables For those test methods that have extremely long lists of analytes, a representative number may be chosen using the following criteria for choosing the number of analytes to be spiked. However, the laboratory shall insure that all targeted components are included in the spike mixture over a 2 year period.

A 8.8.2.3 For methods that have 1-10 targets, spike all components

A 8.8.2.4 For methods that have 11-20 targets, spike at least 10 or 80%, whichever is greater

A 8.8.2.5 For methods with more than 20 targets, spike at least 16 components.

A 8.8.2.6 An LCS "in control" shall be defined as one in which no more than 25% of the individual spiked components may be outside their established criteria.

A 8.8.3 Evaluation. Review the LCS to insure that the results are within the acceptance criteria. While the LCS may be determined to be acceptable by definition, it is critical that professional judgement must be used as well in judging how the LCS results reflect on the condition of the analytical system. It may well be that a situation could occur for which 25% or less of the components are out, yet in the experience of the Lead Analyst and/or Section Chief, the particular components reflect a potentially serious system problem. On the other hand special circumstances may dictate by professional opinion of the Lead Analyst and the Section Chief that analyses should proceed even if the LCS is determined to be unacceptable by definition.

A 8.8.4 Action. Investigate the problem, correct any problems found and determine if the best action is to (1) re-analyze the LCS, (2) re-extract the entire associated batch along with a new LCS or (3) it may be determined to proceed with analysis based on special circumstances of the individual situation. If the decision is to proceed with a LCS that is out of control there must be documentation of the process with details and justification of the decision to proceed. For all circumstances of individual components outside their established acceptance criteria, a J flag should be placed on all samples from the associated batch for these individual components. If, for any reason, the LCS indicates that any specific component has performed so poorly that the qualitative analysis is in question for that specific component and there was a decision to proceed, insure that the results for that component in all samples with the associated batch are flagged as "R" for non-detects and "J" for any positive hits.

A 8.9 Surrogates

A 8.9.1 Objective. Surrogates are used to reflect the overall analytical system and method performance in each sample matrix For this reason the components are chosen to reflect the chemistries of the target analytes of the method and for their being unlikely to be naturally occurring in the sample.

A 8.9.2 Criteria. Surrogates must be added to every sample, blank and spike prior to extraction/preparation. Criteria for an acceptable recoveries should be established from historical data, from method requirements, or as specified by SOPs or the LOQAM. Surrogate acceptance criteria for individual samples shall be as below:

A 8.9.2.1 Extractable Organics by GC/MS Surrogates are calculated for recovery relative to the exact same surrogate standard (separate from the calibration standard) that was used to spike all the samples, blanks, etc.

It is important to make analytical performance judgements only on those surrogates that are free from interferences. Surrogates should not be used for performance judgements if interferences or dilutions are necessary such that the recoveries are rendered ineffective. An analysis is considered out of control if both surrogates for either the base neutral or acid fraction are outside the acceptance windows .

A 8.9.2.2 Volatile Organics by GC/MS. Volatile sample methods do not have 'surrogates' in the purest definition in that all 'surrogates' and internal standards are treated alike, that is, they are analyzed through the entire analytical process. In this instance the 'surrogates' would be more accurately referred to as 'system monitoring compounds'. It is important to make analytical performance judgements only on those surrogates that are free from interferences. Surrogates should not be used for performance judgements if interferences or dilutions are necessary such that the recoveries are rendered ineffective. An analysis is considered out of control if any system monitoring compound is outside the acceptance windows.

A 8.9.2.3 Pesticides/PCBs. Surrogates for Pesticides and PCBs are customized for the particular targets and procedure used in the extraction/clean-up, however, most often include one or two surrogates. Surrogates are calculated for recovery relative to the exact same surrogate standard (separate from the calibration standard) that was used to spike all the samples, blanks, etc. It is important to make analytical performance judgements only on those surrogates that are free from interferences. Surrogates should not be used for performance judgements if interferences or dilutions are necessary such that the recoveries are rendered ineffective. An analysis is considered out of control for any pesticide fraction represented by two surrogates if both surrogates are outside the acceptance windows, or for fractions represented by a single surrogate if that individual surrogate is outside the acceptance window.

A 8.9.3 Evaluation. Review the surrogates to insure that the results are within the acceptance criteria and, if not, that appropriate action is taken.

A 8.9.4 Action. Samples with surrogates determined out of control must be evaluated for the appropriate next steps. Often the corrective action will be re-extraction and re-analysis. Alternatively, it may be prudent to report the data with appropriate data qualifier flags. Professional judgement must be used to determine the most appropriate follow-up action and Lead Analysts and

Supervisors should be consulted. If samples are repeated and acceptable results for all surrogates are obtained the data should be reported from this analysis and no flags are necessary. {providing there are no other technical issues requiring qualifier flags such as holding time, etc.}. If the repeats again produce unacceptable results or it is determined that data should be reported from any analysis with results outside acceptance window, flags must be placed on the associated data as denoted below:

A 8.9.4.1 GC/MS Extractables: If both base-neutral surrogates are outside the acceptance windows and are greater than or equal to10% recovery, flag all associated base-neutral target compounds with a J flag. If both acid surrogates are outside the acceptance windows and greater than or equal to10% recovery, flag all associated acid target compounds with a J flag. If any one base-neutral or acid surrogate has a recovery of less than 10% the first action will be to repeat the extraction and analysis. If recoveries are again less than 10% or if there was insufficient sample to repeat the procedure, flag all associated base neutral or acid target compounds not detected with the "R" flag and any positive results with the "J" flag.

A 8.9.4.2 Volatiles. For a system monitoring compound outside the acceptance windows and greater than or equal to 10% recovery, flag all associated target compounds with a J flag. If any system monitoring compound has a recovery of less than 10% the first action will be to repeat the extraction and analysis. If recoveries are again less than 10% or if there was insufficient sample to repeat the procedure, flag all associated target compounds not detected with the "R" flag and any positive results with the "J" flag.

A 8.9.4.3 Pesticides. Depending on the method used, pesticides analysis may have one or two surrogates in the analysis to be represent specific chemical types. For those instances when two surrogates are used, flag all associated target compounds with the J flag if both surrogates are outside the acceptance windows and have a greater than or equal to 10% recovery. For pesticide fractions represented by one surrogate, qualify all associated target compounds with a J flag if the surrogate is outside the acceptance window and has a greater than or equal to 10% recovery. If any surrogate has a recovery of less than 10% the first action will be to repeat the extraction and analysis. If recoveries are again less than 10% or if there was insufficient sample to repeat the procedure, flag all associated target compounds not detected with the "R" flag and any positive results with

the "J" flag.

A 8.10 Internal Standards

A 8.10.1 Objective. Internal standards consist of specific components (usually specified by the methods and/or SOPs) that are spiked into the extracts just prior to the instrumental analysis, therefore are not subjected to the extraction step. Internal standards serve to evaluate the instrumental analysis system only without including the variable of extraction/preparation.

A 8.10.2 Criteria. Internal standards should be spiked into all extracts (or samples in the case of volatiles) prior to analysis. Acceptance criteria should be established from historical data, from method requirements, or as specified by SOPs or the LOQAM.

A 8.10.3 Evaluation. Internal standards must be evaluated after each run. Internal standards outside established criteria indicate a problem with the analysis system.

A 8.10.4 Action. Internal standards that are outside the established criteria indicate an analysis system problem. Investigate and correct the problem and re-analyze the affected sample(s). If the re-analysis continues to present results outside the criteria or it is determined by professional opinion of the Lead Analyst and the Section Chief that the most appropriate action is to report the data from the original run without the re-analysis, all associated data should be flagged with "J" if the internal standard (s) that are problematic are recovered at 10% or greater. If the problem is loss of internal standard and the amount determined is less than 10% of the expected value associated data must be rejected ("R" flag.) If the decision is to continue analyses using internal standard criteria outside acceptance criteria there must be complete documentation of the process with details and justification of the decision to proceed.

A 8.11 Matrix Spikes/Matrix Spike Duplicates

A 8.11.1 Objective. Spikes of reference materials into sample matrices provides an indication of the method performance relative to "accuracy" or "bias" for the specific matrix spiked. When done in replicate there is the additional indicator of "precision". However, due to differences in matrices it is not generally accepted practice to extrapolate this recovery to all samples in an analytical batch. For this reason, matrix spikes are performed by ASB as may be required by data quality objectives for specific projects or as may be required by a designated

method/SOP. If it is determined that a matrix spike is needed and there are no specified components, project leaders must be consulted to insure that those analytes chosen for spiking are applicable to the specific project.

A 8.11.1.1 Matrix spikes components must be carefully chosen to avoid interference with an accurate assessment. Examples if these would be spiking simultaneously with technical chlordane, toxaphene, and PCBs, and for any compounds with co-eluting peaks.

A 8.11.1.2 For those test methods that have extremely long lists of analytes, a representative number may be chosen using the following criteria for choosing the number of analytes to be spiked. Guidance on choosing the number is listed below:

A 8.11.1.3 For methods that have 1-10 targets, spike all components

A 8.11.1.4 For methods that have 11-20 targets, spike at least 10 or 80%, whichever is greater

A 8.11.1.5 For methods with more than 20 targets, spike at least 16 components.

A 8.11.2 Criteria. Criteria for matrix spike recovery will be as established by methods or may be established using historical data of similar matrices. The use and frequency of matrix spikes will be guided by data quality objectives for specific projects. For example, some projects in order to establish method performance for a specific matrix, may require the analysis of several replicates of matrix spikes into the matrix in question in order to provide a clearer demonstration of method performance on that matrix. This could be especially useful when performed in the beginning of a long term monitoring effort for a specific matrix. After establishing initial performance data follow-up spikes could provide a measure of the ongoing performance over the length of the study.

A 8.11.3 Evaluation. When matrix spikes are performed they must be evaluated against expected recoveries as may be established by methods, SOPs, historical data, or as may have been established for a specific project .

A 8.11.4 Action. Due to the specificity of information gathered by matrix spike data, each instance of “unacceptable” recovery or precision must be thoroughly evaluated by Lead Analysts and Supervisors. In all instances of results outside expected criteria data for the sample spiked, qualifier flags must be placed, at a

minimum, on the affected target analytes for the sample spiked. If there is a further indication that an entire "chemical group" (such as base-neutral or acid components) may be affected then all compounds of the group should be flagged. If it is determined that the matrices of the entire batch are essentially identical it may be appropriate to flag associated samples as well. Affected components should be flagged with "J" if the recoveries are 10% or greater. If the problem is loss of the spiked component and the recovery is less than 10% of the expected value, associated data must have the "R" flag for non-detects and "J" flag for positive hits.. Clear documentation of actions is required.